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Inelastic Stress Relaxation in Single Crystal SiC Substrates

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Abstract. Optical methods were used to measure thermal deformations in commercial 4H- and 6H-SiC wafers. In general, during thermal excursions to 500 ^{o}C, the radii of curvature of the wafers increased (i.e., the wafers became less bowed). Upon cooling to room temperature, nearly all the wafers retained the high temperature radius of curvature values, a characteristic of thermoplastic deformation. Further cyclic excursions to 500 ^{o}C did not yield any significant changes in the radii of curvature, thus signifying the irreversibility of the thermal deformation. The magnitude of the relieved stress following thermal deformation at 500 ^{o}C was estimated to be up to 2.14 MPa. Also observed was a significant difference in the thermoplastic deformation between the off- and on-axis wafers of both polytypes, with the former having more deformation ($\Delta R > 3$ m) than the latter ($\Delta R < 2$ m). An on-axis n-type 6H-SiC that was subjected to thermal cycling to 900 ^{o}C in vacuum also exhibited thermoplastic deformation, with an activation energy of 3.14 ± 0.8 eV and a relieved stress magnitude of 5.67 MPa.

Introduction

Internal stresses in semiconductor substrate materials have over several years been recognized to be deleterious to the yield and reliability of electronic devices and have remained a major research topic in silicon and the III-V semiconductors [1]. The rapid acceleration of bulk crystal growth technology of single crystal silicon carbide (SiC) semiconductors toward high volume commercialization was motivated by the superior electronic properties (i.e., high breakdown fields, wide bandgap, and high thermal conductivity) over traditional semiconductors. These properties are expected to allow SiC based devices to support high speed and high voltage switching, and to operate in much higher temperature and radiation environments than conventional devices. However, it had been asserted that the stress level that develops during the bulk growth of single crystal SiC could be high enough to introduce structural defects such as dislocations (i.e., edge, screw, basal plane dislocations and micropipes) [2]. Variants of these dislocations are known to be responsible for detrimental SiC device performance. Most recently, the forward current-voltage characteristics of 4H-SiC PiN diodes were shown to degrade over time due to the generation of basal plane confined stacking faults (SFs) [3]. Also recently, SFs were shown to be generated in highly doped n-type 4H-SiC when subjected to thermal treatment at 1150 ^{o}C in either oxygen or inert ambient [4], which had been suggested to be due to the expansion of pre-existing quantum well-like dislocations by thermally generated carriers [5]. A few efforts have been reported to understand the stress levels in SiC wafers by numerical simulations, finite element, and empirical modeling [6-8]. However, little effort has been reported to quantify the residual stresses that exist in SiC wafers as a result of commercial production processes (boule growth conditions, wafer sawing, lapping and polishing processes).

In this work, a direct measurement of the thermal deformations in terms of changes in radius of curvature, R, of 4H- and 6H-SiC wafers was performed in-situ by an optical method. The measured thermal deformation allowed for a quantitative evaluation of the residual stresses in the wafers that were introduced during the production process. The results allowed for a correlation between stress and the generation of dislocations in the bulk crystal during growth.

Experimental

Several Si-face, (0001)-oriented n- or p-type, and on- or off-axis 4H- and 6H-SiC 2-inch diameter wafers were commercially procured. The radius of curvature, R, of each as-received wafer was measured with a temperature controlled film stress measurement system, which employs an optical measurement technique whereby a beam from a laser diode scans across the wafer at an angle. The reflected beam undergoes a secondary reflection by a mirror and is detected by a precision position detector [9]. By scanning the diameter of the wafer while the reflected beam is continuously detected, the radius of curvature of the wafer along that diameter is obtained. The wafer was placed in the heating chamber freestanding on molybdenum tripods with tips that provide a very small contact surface area. These tripods extend about 2 mm out of the plane of the heating plate. Subsequently, the wafer was gradually heated from room temperature by 2 ^oC/min to 500 ^oC as it was continually scanned to determine R. The closeness of the wafer to the heating plate and the slow heating process helped to minimize thermal gradients and ensured uniform heating of the 2-inch wafer. This process continued as the wafer was cooled down back to room temperature. The thermal deformation history of the wafer is thus recorded in-situ. Extended thermal cycling of randomly selected SiC wafers was also performed between room temperature and elevated temperatures up to 900 ^oC.

Results and Discussions

Prior to thermal treatment, all the as-received wafers exhibited various degrees of concavity, likely due to the residual stresses introduced during the commercial wafer production process. In almost all cases during thermal excursions to 500 ^oC, the concavity of the wafers decreased (i.e., the wafers became flatter), as shown in the representative graphs of Figs. 1 (a,b). The increase in the radius of curvature was an indication of thermal deformation, generally considered to indicate the gradual relief of the residual stresses in the wafers. It can be observed that upon cooling to room temperature, the radius of curvature usually remained close to the value at 500°C (within limits of measurement error), a

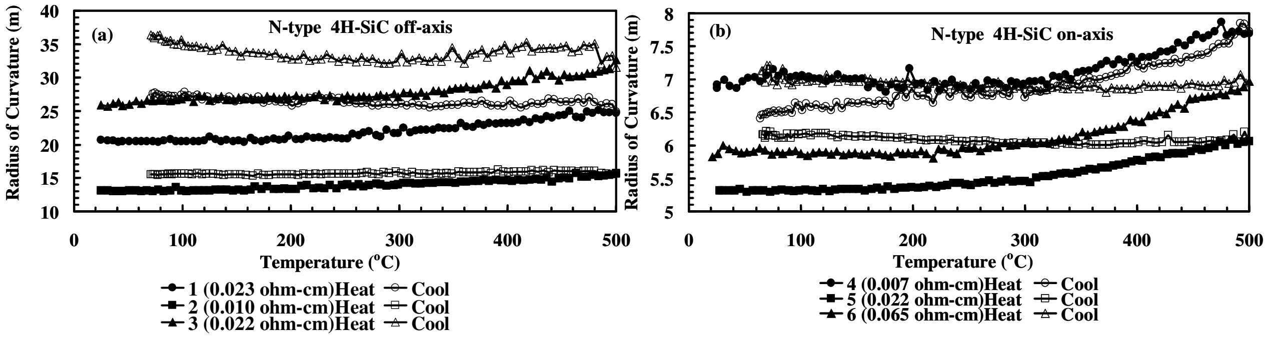

Fig. 1: Representative plots of the radii of curvature as functions of temperature for a) off-axis and b) on-axis n-type 4H-SiC wafers, depicting the irreversible thermal deformation.

classic manifestation of thermoplastic deformation. Repeated cyclic excursions of these wafers to 500 ^oC did not yield any further significant changes in the radius of curvature, thereby confirming the irreversibility of the deformation. From Fig. 1, the following observations are made: The radii of curvature of the as-received off-axis wafers (Fig. 1a) were all found to be consistently higher than those of the on-axis wafers (Fig. 1b). Also, the off-axis wafers exhibited larger changes in R ($\Delta R > 3$ m) than the on-axis wafers ($\Delta R < 2$ m).

The initial radius of curvature, R_o, is believed to be the result of the cumulative stress induced by the thermal gradients that existed during boule growth, sawing of the wafers, and lapping and polishing. This stress, σ_o, can be expressed as [10]:

$$\sigma_o = 0.5 E d R_o^{-1} \tag{1}$$

where E=Young's modulus (Pa), d=wafer thickness (m). This principle can be applied for the static condition under consideration (i.e., absence of thermal transient, strain rate, or emissivity) to calculate the relieved stress via thermal deformation by taking the difference between the inverse of the new radius of curvature after cooling from 500 ^oC, $R_{500\,^oC}$, and the initial curvature, i.e. $1/R = (1/R_{500C} - 1/R_o)$ (m^{-1}). A summary of the measured change in curvature and the calculated relieved stresses of all the wafers investigated are presented in Table 1. It further shows that off-axis wafers consistently exhibited larger initial radii of curvature and experienced more thermal deformation than the on-axis wafer samples (compare R_o and R_{cool}). This is a very significant observation in the light of the previous suggestion by Twigg et al. [11] that the residual stress would be less in the on-axis cut SiC crystals than in the off-axis wafers. Although some of the on-axis wafers experienced low levels of stress relief, it is instructive to note that the low radii of curvature of these wafers (larger bows than in off axis wafers), even after thermal treatment, is indicative of high residual stresses still trapped in them. Therefore, it is yet unclear to what extent these differences in thermal deformations are orientation dependent since no published studies are known to exist and this will be subject to future investigation. It can also be seen that within the range of the doping levels in these wafers, no correlation was found between the thermal deformations and doping level. This is consistent with recently published results that showed little change in the lattice constant of 4H-SiC different nitrogen concentrations [12]. The resulting magnitude of the relieved stress following thermoplastic deformation at 500 °C was estimated to be as high as 2.14 MPa (see Table 1). Numerical modeling by Bakin et al. had predicted that the stress in 50 mm diameter, 20 mm thick boules ranges between -2 MPa to 18 MPa [6]. Müller et al. resolved the shear stress values in physical vapor transport grown SiC boules and found they were as high as 55 MPa at the bottom to 2 MPa at growth front [7]. For the R values obtained at 500 °C, the range of stresses calculated from the thermal deformation measurements in this work agrees very well with these published values.

Sample	Polytype/ Orientation	ρ (Ω-cm)	R_o (m)	R_{cool} (m)	1/R (m^{-1})	σ_{500C} (MPa)
1	4H/n-type/8°	0.022	20.72	26.92	-0.011	1.14
2	4H/n-type/8°	0.01	13.17	15.56	-0.012	1.28
3	4H/n-type/8°	0.022	22.66	36.37	-0.017	1.86
4	4H/n-type/0.2°	0.007	6.87	6.41	0.01	-0.71
5	4H/n-type/.05°	0.022	5.32	6.16	-0.027	1.80
6	4H/n-type/.17°	0.065	5.83	7.13	-0.031	2.14
7	6H/p-type/3.5°	1.7	19.11	22.82	-0.01	0.91
8	6H/p-type/3.5°	2.3	14.6	18.01	-0.01	1.32
9	6H/p-type/3.3°	2.7	16.12	23.73	-0.012	2.11

Table 1: Summary of changes in radius of curvature of the on- and off-axis 4H- and 6H-SiC substrates after thermal excursions to 500 °C and cooling. The calculated thermal stresses corresponding to the thermal deformation are also shown.

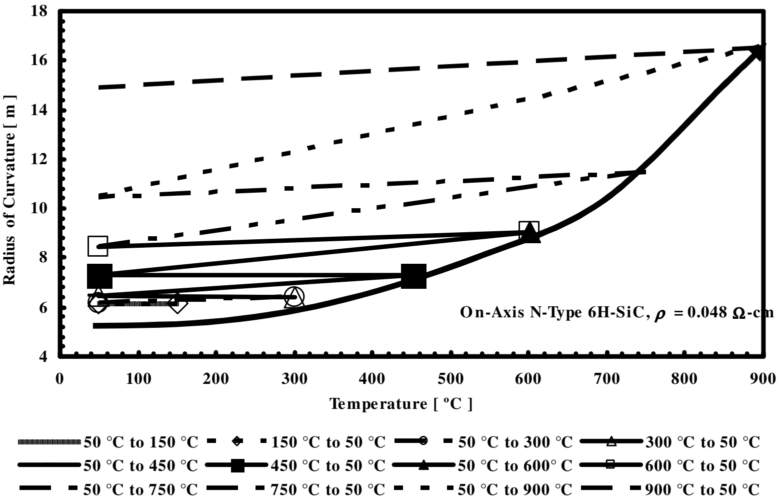

Fig. 2: Representative cyclic thermal history of an on-axis n-type 6H-SiC wafer showing the irreversible changes in radius of curvature at every temperature step. Further increase in temperature shows a corresponding change in curvature.

In order to determine the nature of the thermoplastic deformation beyond 500 °C, cyclic thermal treatment was extended to 900 °C (in vacuum) at 150 °C intervals. The temperature was first cycled twice between room temperature and 150 °C. Then two more cycles were performed between room temperature and 300 °C, and so on. In all instances, the values of the radius of curvature measured during the second cycle to a given temperature were the same as for the first. Fig. 2 shows the changes in the radius of curvature as a function of temperature. Each time the temperature was extended higher, further deformation was induced and with a corresponding value of relieved stress. For this on-axis 6H-SiC wafer annealed up to 900 °C, the calculated relieved stress was 5.67 MPa. Extrapolation to growth temperatures of recent deformation experiments by Fujita et al. have shown that basal dislocations are created at much lower shear stresses (of the order of a few kPa), with an activation energy of 3.4 ± 0.7 eV [13]. In this experiment, the activation energy of deformation was estimated to be 3.14 ± 0.8 eV, which was obtained between 450 ^{o}C and 900 ^{o}C.

Conclusion

The results from this work lead to the following conclusions. For as-received commercial 4H- and 6H-SiC wafers, larger radii of curvature (lower bow) exist in off-axis wafers than in the on-axis wafers (larger bow). It was also observed that the off-axis wafers exhibited characteristically higher thermoplastic deformations than the on-axis wafers. These measurements also provide evidence that because of the tilt of the basal planes in commercial SiC wafers, significant shear stresses arise that may cause the generation of SFs. Thus, it is possible that the SFs observed in many recent experiments on off-axis 4H-SiC wafers are due to thermal stress induced motion of leading partial dislocations that were introduced in the 4H-SiC during boule growth. Thermoplastic deformations resulting in changes in the radius of curvature were found to be independent of the doping level or conductivity (p- or n-type).

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